![Interreg North-West Europe / NWE-REGENERATIS logo — European Regional Development Fund]

# D. I2.1.2. SITE SPECIFIC REPORT ON TRADITIONAL SAMPLING INVESTIGATIONS ON POMPEY SITE (FR)

P. Kessouri[1], C. Ryckebusch[1], L. Ardito[1], N. Cros[1], B. Mignon[2]

[1]BRGM, [2]CTP

# SUMMARY

# 1 LIST OF FIGURES

# 2 LIST OF TABLES

# 3   INTRODUCTION

Pompey is one of the three pilot sites of the NWE-REGENERATIS project. It is a former tailing pond owned by the EPFGE (Etablissement Public Foncier de Grand Est, Public Real-Estate Company of Grand Est region). The site has been chosen for two main reasons: (1) it hosted various activities for iron based alloys production; (2) it was just rehabilitated on surface, and historic documentation and investigations are done with respect of the French legislation and threshold values. One of the interest of this site is that it allows testing the REGENERATIS methodologies developed within WPT1 and WPT2 on a site that was already been remediated.

Site works provided access to material to perform lab trials and also allowed on-site geophysical measurements. Two phases of sampling were handled on site: (1) traditional sampling prior to the geophysical investigations; (2) targeted sampling after the geophysical investigations.

This site-specific report is intended to provide specific data on pre- and post- sampling investigations including: (1) a sampling plan including the general methodology applied to the site and the type of sampling chosen; (2) a list of samples taken and characterization performed in laboratory; (3) a table summarizing the results.

# 4   PRESENTATION OF THE POMPEY SITE

The Pompey site is a former tailing pond from the iron and steel complex of Pompey-Frouard-Custines, located 10 km North from Nancy. The steel complex was active from 1870 to 1986. It is renowned for producing cast iron and special steels, such as ferromanganese (ferro-alloy rich in manganese). The last blast furnace of the Pompey-Frouard-Custines iron and steel complex was stopped in 1986. Over time, a forest ecosystem developed on the former tailing pond. The dike delimiting the site was planted with a curtain of black locust trees in 1997. The rest of the pond gradually got covered with diversified deciduous vegetation, more or less dense depending on the area.

The geological substratum of the former tailing pond consists of the Lias marl formations (at 181 m NGF), which are covered by alluvium from the two rivers, composed of coarse siliceous materials (sands, gravel and pebbles) at the base over 3 to 6 m surmounted by finer materials (sands, silts and clays) on 1 to 3 m. These alluvial formations were locally exploited and backfilled with waste rock and iron and steel by-products.

The depth of the deposits in the basin is estimated at around 10 m. The surface of the former pound is estimated to 26 000 m$^2$, for a total estimated volume of wastes equal to 260 000 m$^3$.

The waters of the alluvial table would circulate from the channeled Moselle towards the Meurthe, whose level is lower. The piezometric levels measured in 2002 are 187.5 m NGF upstream (South-West of the island) and 184 m NGF downstream (East of the island), the basin surface being at 197 m NGF (ANTEA, 2002).

# 5   OBJECTIVES OF THE SAMPLING INVESTIGATIONS

Traditional sampling investigations on past metallurgical sites and deposits aims at characterizing the nature, physical and chemical composition of the wastes at a punctual location. On the other hand, geophysical methods allow mapping the variations of geophysical properties that are indirectly linked to the physical and chemical nature of the formations in place, over the entire surface of the deposit. The combination of the traditional sampling investigations and the geophysical imaging will allow interpreting in a semi-quantitative approach the geophysical observations, using the chemical and physical laboratory characterizations of the punctual samples.

Two conventional sampling campaigns were run at the Pompey pilot site (see investigation plan - deliverable DI2.1.2):

The first one, prior to the geophysical investigations, took place in the fall 2020. A pit is already digged since 2010 on site for the first 2 meters of soils (see "fosse" in Figure 1). Without other complementary information, the tailing pond is supposed to contain horizontal layers of soils with composition variations being only vertical and not lateral. The first samples of soils to analyze were thus taken within the pit, in the first two meters. pXRF and XRF analysis were run on samples extracted from the trench to characterize their chemical composition.

The second one, posterior to the geophysical investigations, took place in the summer 2021. Five soundings were led at locations determined using the interpreted geophysical measurements. Samples were taken from 0 to 9 m deep. pXRF analysis, as well as physical characterization (for one location) were run on samples extracted from the trench to characterize their chemical composition.



*Figure 1 : Map of the different sampling locations on the Pompey site.*

The goals of the sampling investigations were to: (1) characterize variability in the composition of the site and (2) allow semi-quantitative interpretation of the geophysical measurements

# 6 CHARACTERIZATION METHODS

## 6.1 X-RAY FLUORESCENCE SPECTROMETERS

Two types of spectrometers were used to analyze the chemical composition and concentrations of the different samples:

- A portable X-ray fluorescence spectrometer was used at BRGM to characterize most of the samples after drying and sieving steps
- A more detailed analysis using a more accurate laboratory X-ray fluorescence spectrometer was used at CTP to characterize two selected samples after several separation procedures

## 6.1.1 Characterization using the portable X-ray fluorescence spectrometer

### 6.1.1.1 Presentation of the pXRF instrument used

The pXRF used is a NITON® XL3t980 (see Figure 2). It is equipped with a 50 kV X emitter tube and a GOLDD+ detector (Geometrically Optimized Large Area Drift Detector), with soil and ore modes. This device allows a multi-elemental analysis. It can detect or analyze the elements of atomic number between sulfur and uranium, provided that their content is sufficient and that there is no interferences.



Figure 2 : portable X-ray fluorescence spectrometer (XL3t980 model)

The two analytical modes correspond to a specific calibration of the spectrometer, adapted to the content ranges to be measured. The X-ray fluorescence response is not completely linear over the wide range of contents covered. We consider here as high:

- contents higher than 1% for the elements usually in traces;

- contents greater than 10% for the major elements when a particular abundance is likely to affect the measurement of the other elements by a matrix effect.

The measurement in "ore" mode is necessary when high levels are expected to be found in materials other than soils, whereas the "soil" mode is generally used for the determination of the pedogeochemical background which corresponds to low values in soils.

Measurements are made in ore mode after internal calibration. The counting time is 30 s for each of the 4 filters (NITON®), which makes a total time of about 120 s.

Even if these devices are calibrated, there is a bias between field analyzes and X-ray laboratory analyzes such as ICP spectrometry. Indeed, in pXRF, the device analyzes a volume containing the material to be analyzed but also organic matter, water and air. The more these three compounds are present, the less material there is. It is therefore logical that the contents obtained in wit pXRF are lower than those obtained by ICP spectrometry after drying, grinding and mineralization in acid of the samples. Depending on the state of the samples (composition and preparation), these biases are more or less significant.

### 6.1.1.2 Preparation of the samples:

All the samples analyzed are dried at 105°C for 24 hours, weighted, and then dried at 105°C again and weighted, until the weigth is stabilized, indicating that the sample is dry.

All the samples are then sieved at 2 mm to avoid the presence of coarse material during the pXRF measurements. Some parts of the samples are also crushed, when the presence of clods/agglomerated are observed.

A small sample holder is used to place the samples before the measurements. The surface of the samples is made as flat as possible to decrease the measuring noise. The pXRF analysis is run directly on the sample, without adding a protective cover.

Several elements have concentrations that are lower than the limit of detection (LOD) : Mo, Co, Ni, Au, Cl, Se, Pd, W and Bi.

The contents of the light elements (Mg, Al, Si, P and S) are only indicative because the preparation of the samples is not the best to quantify them correctly, it is absolutely necessary for these elements that the samples are ground bellow 100 µm.

The peaks linked to As and Pb are very close to each other in the spectrum. The concentration of As estimated is probably false, because of the high Pb concentrations observed.

## 6.1.2 Semi-quantitative characterization using laboratory X-ray fluorescence spectrometer

### 6.1.2.1 Presentation of the XRF instrument used

Each particle size fraction was analysed individually by X-ray fluorescence spectrometry (XRF) to determine its elemental chemical composition. The instrument used is an WDXRF S8 TIGER spectrometer from Bruker.

The XRF spectrometry method is a relatively effective quantitative technique to determine the elemental composition of any material, with trace element abundances at the ppm level. Due to instrumental limitations and low X-ray yields for the light elements, it is often difficult to quantify elements lighter than sodium.
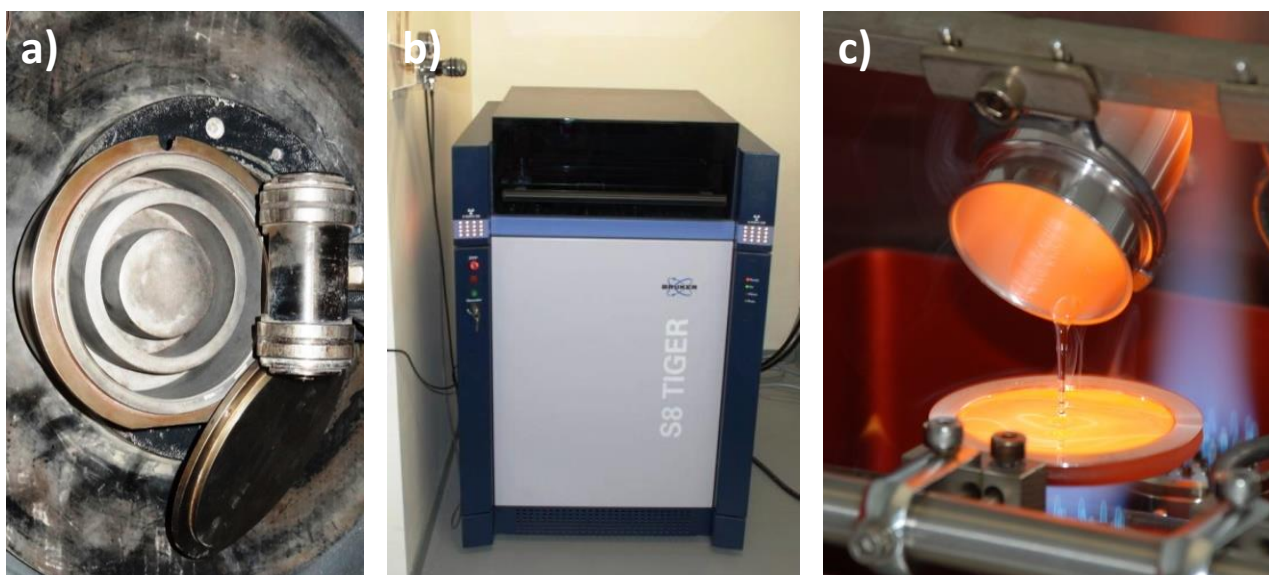


Figure 3 : a) Ring mill used to ground the samples. XRF device (b)) on fused bead (c)).

### 6.1.2.2 Preparation of the samples

Before the X-ray analysis, the two samples analyzed by CTP are also following two mineral processing steps (see Figure 4): (1) sieving and (2) magnetic separation.

Sieving:

All the samples are first screened through a 1 cm mesh after a first dry (105°C) in order to remove exogenous material such as stones, wood, organic matter, that are not part of the PSMD deposits. Given the fineness of the material, the sieving takes place in two stages:

- a first wet sieving at 106 microns to remove fine particles agglomerated or adhering to coarser particles;
- a dry sieving on particles larger than 106 microns and previously dried at 105°C at different mesh sizes: 106 µm, 600 µm, 1.18 mm and 2.36 mm

Magnetic separation:

For each particle size range previously defined, magnetic separation is carried out in two steps:
- a first manual passage through the ferrite bar (2000 Gauss), allowing to remove the highly magnetic fraction
- a second pass over a more powerful magnetic bar (6000 Gauss), allowing to separate a more slightly magnetic fraction from the rest of the non-magnetic material.
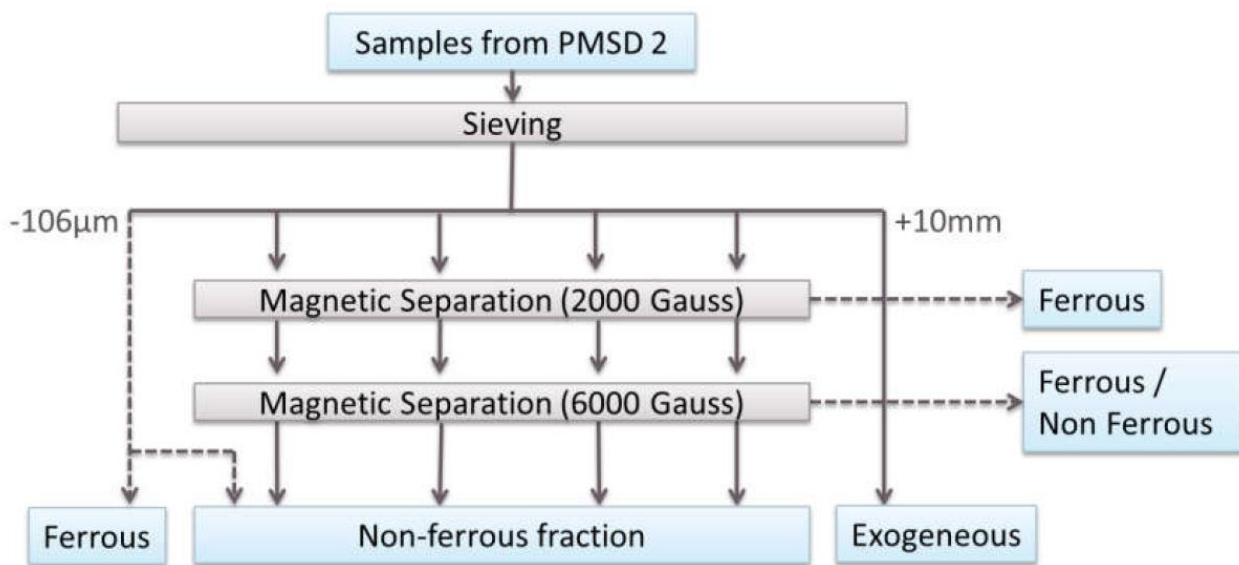


*Figure 4 : Flow-sheet of the process applied on both Pompey samples*

A semi-quantitative XRF analyses is realized on samples from each separated magnetic fraction, for each particle size range. Before the X-ray analysis, each sample is ground in a ring mill to get a very fine powder which is then prepared as a bead using 1 g of material mixed with fluxes (lithium metaborate and tetraborate).

# 7 PRE-SAMPLING INVESTIGATIONS

## 7.1 SAMPLING ON SITE

Pre-sampling investigation, prior to the geophysical investigations, took place from the 23rd to the 25th of November 2020. A pit is already dug since 2010 on site to investigate the first 2 meters of soils (see "fosse" in Figure 1). The first samples of soils to analyze were thus taken within the pit, in the first two meters (see Figure 5). Two types of samples were extracted:

- 17 soil samples were taken every 20 cm from 0.2 m to 1.8 m depth. Two different sides of the pit were investigated (noted P2 and P4) for portable X-ray fluorescence spectroscopy analysis at BRGM: 9 samples for P2 and 8 samples for P4. Each soil sample weights in average 330 g.

– 2 soil samples were taken in the pit at 2 depths: [1.0 - 1.1] m and [1.65 - 1.80] m for deeper laboratory analysis at CTP's center in Tourney. The choice of these two depths were dictated by preliminary soil analysis from Huot (2013) that detected higher content of Pb and Mn at 1 m; and of Zn at 1.8 m (also see appendix 1)
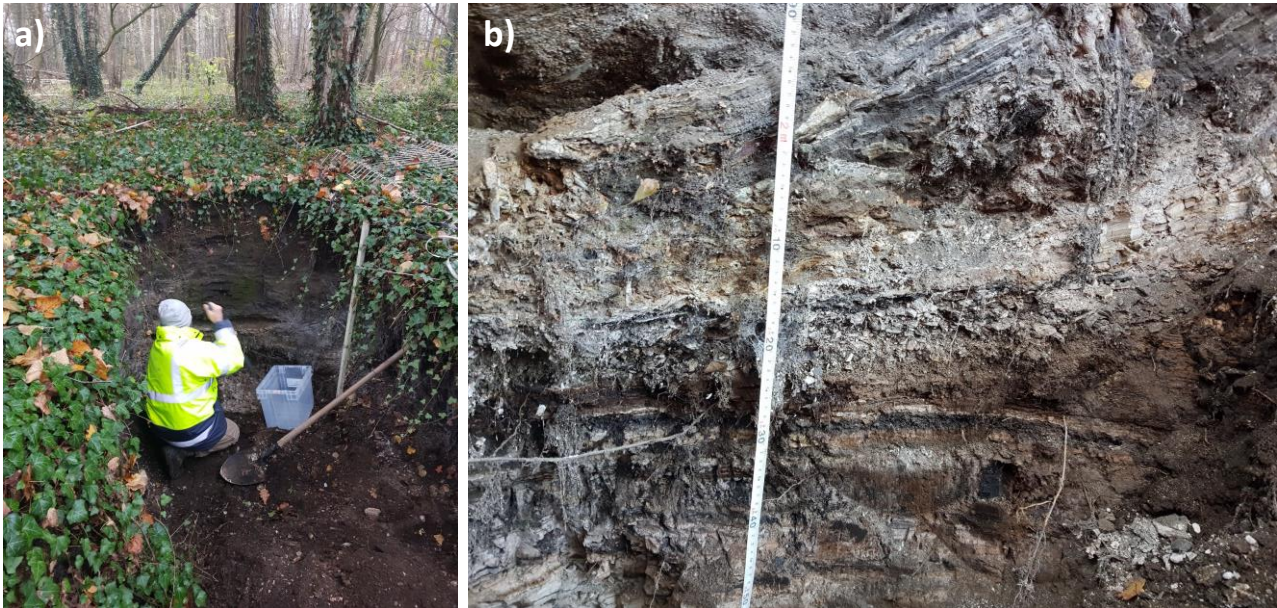


*Figure 5 : a) extraction of soil samples from the trench pit; b) zoom on the variation of techno-soil layers observed within the trench pit*

## 7.2 LABORATORY CHARACTERIZATION

### 7.2.1 Mineral processing

Each of the two samples analyzed at CTP was studied separately using mineral processing separation techniques (see deliverable DT2.1.1) : The first sample **S1** was taken between 1,0 to 1,1 m depth (ref. M 10265 A) and the second sample **S2** was taken between 1,65 and 1,80 m depth (ref. M 10265 B).

#### 7.2.1.1 Sieving

The sieving methodology used in this study is described in section 6.1.2.2. Taking into account the nature of the sample (settling sludge), a first dry screening at 10 mm was carried out in order to remove exogenous (stone, wood, organic matter), theoretically free of metals. The fraction smaller than 10 mm was then screened at different mesh sizes to produce several granulometric sections, with a double objective:

- reducing the gap in particle size to improve the separation efficiency of downstream separation techniques;
- concentrating some metals in specific particle size ranges (heavy metals preferentially found in fines) in order to reduce subsequent treatment costs.

The different particle size range are:

– < 106 µm
– 106 µm - 600 µm
– 600 µm – 1.18 mm
– 1.18 mm – 2.36 mm
– 2.36 mm – 10 mm
– > 10 mm

Particle size distribution results are shown in Figure 6. The sample S2 taken at 1.8 m is composed largely of coarse particles (greater than 10 mm) whereas the surface sample was composed mainly of fine particles (below 600 µm).

**a)**

| Particle size | S1 - M10265A | S2 - M10265B |
|---|---|---|
| | Cumulative refusal | Cumulative refusal |
| (µm) | (%) | (%) |
| 10 000 | 15,3 | 45,1 |
| 2 360 | 18,9 | 46,7 |
| 1 180 | 21,4 | 47,3 |
| 600 | 28,4 | 49,1 |
| 106 | 73,7 | 58,1 |
| 0 | 100,0 | 100,0 |

**b)**
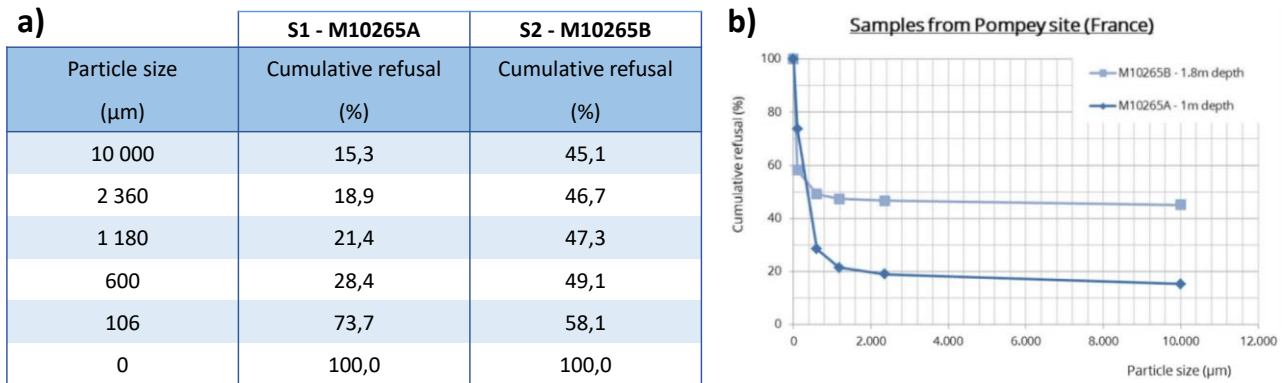


*Figure 6 : Sieving results: Particle size distribution of both samples (S1 at 1 m deep and S2 at 1.7 m deep) from the pit trench represented as: a) a table; b) a graph*

### 7.2.1.2 Magnetic separation

Given the steelmaking history of the Pompey site, the presence of ferrous residues with a high magnetic susceptibility is more than possible. A magnetic separation step was therefore carried out on each granulometric fraction (under 10 mm) of each sample, except for the fraction below 106 µm. In fact, below this particle size, every particle, including non-magnetic one, is attracted by electrostatic effect.

The tests were carried out manually using magnetic rods. A first passage through the ferrite bar (2000 Gauss), simulating a treatment with an Overband, was carried out, which allowed the removal of the highly magnetic fraction. A second pass over a more powerful magnetic bar (6000 Gauss), simulating the use of a magnetic drum, separated a more slightly magnetic fraction from the rest of the non-magnetic material.

The mass balance coming from both magnetic separation tests is shown in Tableau 1. It can be seen that the sample S1 taken at 1.1 m (M 10265 A) has a much higher proportion of magnetic particles than the deeper sample S2 (M 10265 B). In general, the relative quantity of magnetic elements is higher in the finer grain size fractions whatever the sample.

*Tableau 1 : Mass balance of the magnetic separation tests on samples S1 and S2*

| S1 | | | Magnetic separation | | | | | |
|---|---|---|---|---|---|---|---|---|
| Particle size (µm) | | Weight | Highly magnetic | | Medium magnetic | | Non magnetic | |
| min | max | (g) | (g) | (% per size) | (g) | (% per size) | (g) | (% per size) |
| 106 | 600 | 916.5 | 497.7 | 54% | 250.6 | 27% | 142.8 | 16% |
| 600 | 1 180 | 142.6 | 55 | 39% | 54.2 | 38% | 33.4 | 23% |
| 1 180 | 2 360 | 50.2 | 13.5 | 27% | 18.9 | 38% | 17.8 | 35% |
| 2 360 | 10 000 | 73.4 | 31 | 42% | 20.3 | 28% | 22.1 | 30% |
| **106** | **10 000** | 1182.7 | 597.2 | 50% | 344 | 29% | 216.1 | 18% |

| S2 | | | Magnetic separation | | | | | |
|---|---|---|---|---|---|---|---|---|
| Particle size (µm) | | Weight | Highly magnetic | | Medium magnetic | | Non magnetic | |
| min | max | (g) | (g) | (% per size) | (g) | (% per size) | (g) | (% per size) |
| 106 | 600 | 108.3 | 25.4 | 23% | 20.1 | 19% | 62.8 | 58% |
| 600 | 1 180 | 20.9 | 0.9 | 4% | 4.9 | 23% | 15.1 | 72% |
| 1 180 | 2 360 | 7.7 | 0.3 | 4% | 1.7 | 22% | 5.7 | 74% |
| 2 360 | 10 000 | 19.1 | 0.4 | 2% | 1.5 | 8% | 17.2 | 90% |
| **106** | **10 000** | 156 | 27 | 17% | 28.2 | 18% | 100.8 | 65% |

### 7.2.1.3 Conclusions

Based on the mineral processing analysis on the samples S1 and S2, differences can already be highlighted:

- At 1.1 m deep, the techno-soil is composed mainly of fine particles (bellow 600 µm), and has a much higher portion of magnetic particles, revealing the potential presence of ferrous residues
- At 1.7 m deep, the techno-soil is composed mainly of coarse particles (greater than 10 mm), and within its finer portions, has a lower proportion of magnetic particles, indicating a potential lower proportion of ferrous residues.

## 7.2.2 Chemical analysis

### 7.2.2.1 pXRF results

The results obtained for the pXRF analysis of the 17 samples are shown in Figure 7. The concentration trends observed are very similar between the two profils P2 and P4, showing that: (1) the results are reliable and (2) the lateral variations of soils within the pit are low compared to the vertical variations. Three main layers can be observed within the trench pit:

- – <u>from 0 to 0.8 m:</u> the concentration of Fe is stable, varying between 20 % to 30 %, while Mn is ranging between 2 and 3 %, Pb between 0.3 and 0.7 %, and Sn around 0.1 %
- – <u>from 0.8 to 1.4 m:</u> the concentration of Fe is decreasing from 20 % to 3 % with depth, while Mn drops to 1 %, Pb increases from 0.7 % to 4 %, and Sn increases to reach a maximum of 1 % at 1.4 m deep
- – <u>from 1.4 to 1.8 m:</u> the concentration of Fe (ranging from 2 % to 10% maximum) is lower than in the first two layers of the pit. The concentration of Mn is varying between 7 % to 20 %. The concentration of Pb is the highest in this lower layer (between 1 and 5 %), and Sn as well (betwwen 0.4 % and 1 %).

We note that the top layer (above 0.8 m) is more concentrated in Fe, while the bottom layer is more concentrated in Mn. Iron and manganese are apparently not bound together, and are thus not present in their alloy forms.

It can also be noted that the values for Pb, Mn and Zn measured in Huot (2013) are not found back in this study. Indeed, the peak concentration of Pb and Mn at around 1 m deep, measured at respectively 12% and 35% in Huot (2013), is found equal to respectively 0.7 % and 1 % in this study.
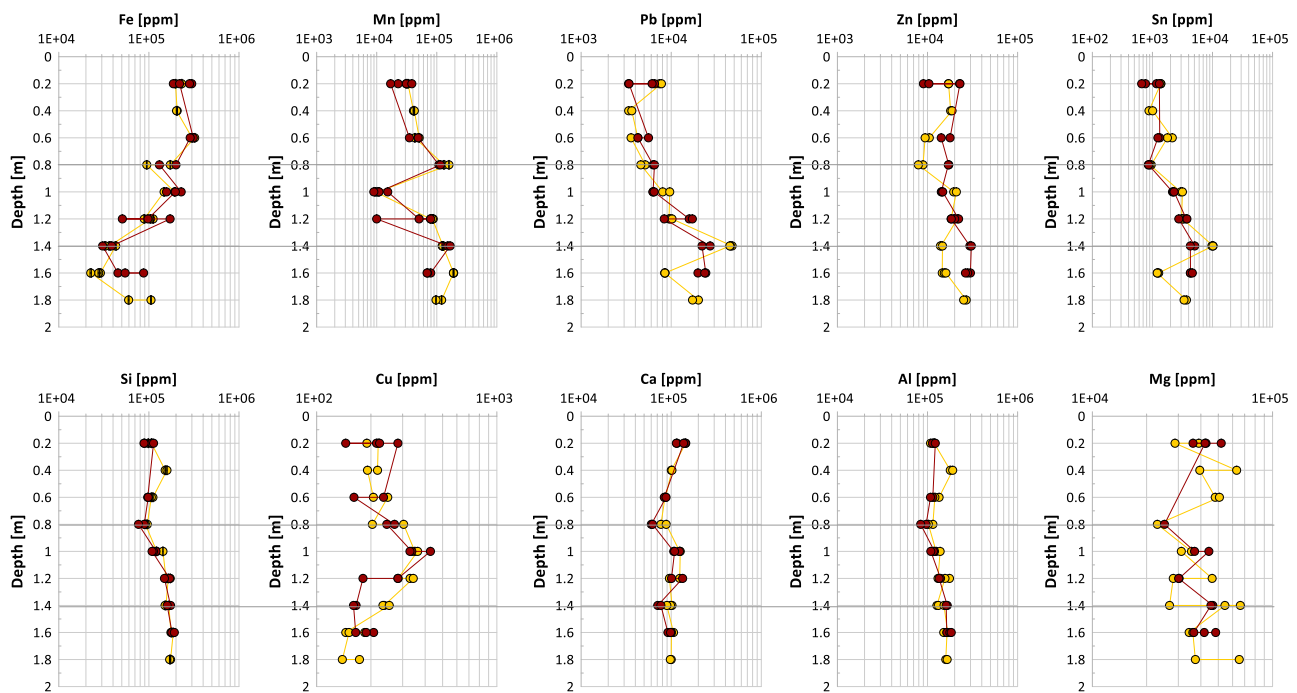


*Figure 7 : Variation of concentration of different selected chemical elements versus depth for two different soil profiles investigated within the trench pit (P2 in yellow and P4 in red)*

### 7.2.2.2  XRF results

For the two samples analyzed by CTP, a complete chemical analysis was carried out on each separated fraction in order to determine the potentially recoverable metal contents. Semi-quantitative XRF analyses were realized on pearls from each magnetic fraction for each particle size range.

#### Average results

The composition of both samples sieved at 10 mm was recalculated based on the mass balance of the different fractions and their respective analyses (see Tableau 2).

We note that the sample S1 taken at 1.1 m depth is more concentrated in iron and sample S2 from 1.7 m is more concentrated in manganese. Therefore, it seems that iron and manganese are not bound together, contrary to what is expected for Fe/Mn alloys.

It can also be noted that the values for Pb, Mn and Zn measured in Huot (2013) are not found back in this study. Indeed, the peak concentration of Pb and Mn at around 1 m deep, measured at respectively 12% and 35% in Huot (2013), is found equal to 0.8 % and 1.2 % in this study. These values are very close to the one measured using the pXRF characterization described above.

*Tableau 2 : Semi-quantitative XRF analytical results on recombined fractions below 10 mm for the two Pompey samples S1 and S2*

| Element | S1 | S2 | Element | S1 | S2 |
|---|---|---|---|---|---|
| Fe (%) | 21,8 | 5,9 | Ti (%) | 0,07 | 0,01 |
| Ca (%) | 13,2 | 11,4 | V (%) | 0,02 | 0,00 |
| Si (%) | 6,8 | 9,5 | Sr (%) | 0,05 | 0,06 |
| Al (%) | 3,5 | 4,9 | Cu (%) | 0,04 | 0,08 |
| Zn (%) | 1,7 | 2,9 | Cr (%) | 0,02 | 0,00 |
| Mg (%) | 1,0 | 1,7 | Ni (%) | 0,03 | 0,03 |
| Mn (%) | 1,2 | 8,9 | Cl (%) | 0,02 | 0,00 |
| P (%) | 0,3 | 0,1 | S (%) | 0,04 | 0,23 |
| Pb (%) | 0,8 | 2,4 | | | |
| K (%) | 0,5 | 0,4 | | | |
| Sn (%) | 0,3 | 0,7 | | | |
| Na (%) | 0,1 | 0,3 | | | |
| Ba (%) | 0,2 | 0,6 | | | |

## Results for each particle size range

The impact of sieving techniques on the potential concentration of metals of economic interest in specific particle size ranges was also estimated. The results detailed in Tableau 3 and Tableau 4 show that, except for iron which concentrates in fractions below 600 microns, there is no significant impact on non-ferrous metals (Zn, Pb, Mn, Sn).

*Tableau 3 : Semi-quantitative XRF analytical results on each particle size range below 10 mm for the sample S1*

| Element | ... | | | | | |
|---|---|---|---|---|---|---|
| | **Sample S1** (1.0-1.1 m depth) | | | | | |
| Element | initial | <106µm | 106-600µm | 600-1180µm | 1,18-2,36mm | 2,36-10mm |
| Fe (%) | 21,8 | 27,7 | 20,6 | 13,1 | 11,1 | 17,1 |
| Ca (%) | 13,2 | 10,0 | 13,1 | 18,1 | 22,2 | 21,3 |
| Si (%) | 6,8 | 7,3 | 6,7 | 6,9 | 5,7 | 4,8 |
| Al (%) | 3,5 | 4,0 | 3,3 | 3,6 | 3,0 | 2,5 |
| Zn (%) | 1,7 | 1,9 | 1,6 | 1,9 | 1,7 | 1,6 |
| Mg (%) | 1,0 | 1,2 | 0,9 | 0,9 | 0,8 | 0,8 |
| Mn (%) | 1,2 | 1,0 | 1,1 | 1,6 | 1,9 | 2,5 |
| P (%) | 0,3 | 0,3 | 0,3 | 0,2 | 0,1 | 0,2 |
| Pb (%) | 0,8 | 1,0 | 0,7 | 1,0 | 0,8 | 0,7 |
| K (%) | 0,5 | 0,4 | 0,5 | 0,6 | 0,5 | 0,4 |
| Sn (%) | 0,3 | 0,4 | 0,2 | 0,4 | 0,4 | 0,3 |
| Na (%) | 0,1 | 0,0 | 0,2 | 0,2 | 0,0 | 0,2 |
| Ba (%) | 0,2 | 0,2 | 0,1 | 0,2 | 0,1 | 0,2 |
| Ti (%) | 0,07 | 0,08 | 0,08 | 0,05 | 0,04 | 0,04 |
| V (%) | 0,02 | 0,03 | 0,02 | 0,01 | 0,01 | 0,01 |
| Sr (%) | 0,05 | 0,04 | 0,05 | 0,06 | 0,08 | 0,07 |
| Cu (%) | 0,04 | 0,06 | 0,04 | 0,05 | 0,06 | 0,04 |
| Cr (%) | 0,02 | 0,03 | 0,02 | 0,02 | 0,02 | 0,02 |
| Ni (%) | 0,03 | 0,03 | 0,03 | 0,03 | 0,03 | 0,02 |
| Cl (%) | 0,02 | 0,00 | 0,03 | 0,02 | 0,04 | 0,03 |
| S (%) | 0,04 | 0,09 | 0,01 | 0,04 | 0,11 | 0,08 |

*Tableau 4 : Semi-quantitative XRF analytical results on each particle size range below 10 mm for the sample S2.*

| Element | | | | | | |
|---|---|---|---|---|---|---|
| | **Sample S2** (1.65-1.8 m depth) | | | | | |
| Element | initial | <106µm | 106-600µm | 600-1180µm | 1,18-2,36mm | 2,36-10mm |
| Fe (%) | 5,9 | 5,2 | 10,3 | 3,2 | 3,1 | 3,1 |
| Ca (%) | 11,4 | 10,2 | 14,8 | 18,1 | 16,4 | 13,0 |
| Si (%) | 9,5 | 10,4 | 6,2 | 6,4 | 7,0 | 10,0 |
| Al (%) | 4,9 | 5,4 | 3,1 | 3,3 | 3,9 | 5,3 |
| Zn (%) | 2,9 | 3,2 | 1,9 | 2,2 | 2,2 | 2,5 |
| Mg (%) | 1,7 | 1,8 | 1,0 | 1,4 | 1,7 | 2,7 |
| Mn (%) | 8,9 | 9,0 | 9,8 | 8,5 | 7,4 | 4,6 |
| P (%) | 0,1 | 0,1 | 0,2 | 0,1 | 0,1 | 0,1 |
| Pb (%) | 2,4 | 2,3 | 3,0 | 2,4 | 2,0 | 2,2 |
| K (%) | 0,4 | 0,4 | 0,4 | 0,3 | 0,4 | 0,4 |
| Sn (%) | 0,7 | 0,8 | 0,4 | 0,5 | 0,4 | 0,6 |
| Na (%) | 0,3 | 0,4 | 0,2 | 0,1 | 0,2 | 0,0 |
| Ba (%) | 0,6 | 0,6 | 0,7 | 0,6 | 0,5 | 0,6 |
| Ti (%) | 0,01 | 0,00 | 0,05 | 0,04 | 0,00 | 0,02 |
| V (%) | 0,00 | 0,00 | 0,01 | 0,01 | 0,00 | 0,00 |
| Sr (%) | 0,06 | 0,06 | 0,07 | 0,07 | 0,07 | 0,06 |
| Cu (%) | 0,08 | 0,06 | 0,16 | 0,13 | 0,13 | 0,06 |
| Cr (%) | 0,00 | 0,00 | 0,01 | 0,00 | 0,00 | 0,00 |
| Ni (%) | 0,03 | 0,02 | 0,06 | 0,06 | 0,06 | 0,02 |
| Cl (%) | 0,00 | 0,00 | 0,00 | 0,00 | 0,00 | 0,00 |
| S (%) | 0,23 | 0,24 | 0,20 | 0,18 | 0,12 | 0,29 |

## Results for different magnetic fractions

The average composition of the recombined magnetic fractions for each sample is shown in Tableau 5. For both samples, the magnetic fraction is mainly composed of iron (28-30%) with various concentrations of other non-ferrous metals.

Manganese is more concentrated in the low-magnetic and non-magnetic fractions even if its grade (between 1 and 12% w/w) remains low compared to that observed in an ore (~40%). This observation confirms that manganese is not in alloy with iron.

In sample S1 (M 10265 A), the composition of the fraction below 106 µm (see Tableau 3) is similar to that of the magnetic fraction over 106 microns so that these two fractions could be recovered together.

In sample S2 (M 10265 B), the behavior is different as the fine fraction (see Tableau 4) has a composition that is intermediate between the low and non-magnetic ones and could be recovered for its manganese content.

*Tableau 5 : Impact of magnetic separation on both samples S1 and S2. 2000 G represents the highly magnetic fraction, 6000 G represents the medium magnetic fraction.*

| Element | S1 initial | S1 (+106µm - 10mm) 2000G | 6000G | Non magn | S2 initial | S2 (+106µm – 10mm) 2000G | 6000G | Non magn |
|---|---|---|---|---|---|---|---|---|
| Fe (%) | 21,8 | 28,1 | 11,7 | 6,9 | 5,9 | 30,4 | 7,8 | 2,4 |
| Ca (%) | 13,2 | 10,9 | 15,5 | 23,1 | 11,4 | 9,1 | 11,1 | 18,8 |
| Si (%) | 6,8 | 6,8 | 7,8 | 5,8 | 9,5 | 5,4 | 7,6 | 6,1 |
| Al (%) | 3,5 | 3,6 | 3,8 | 2,6 | 4,9 | 2,7 | 3,9 | 3,0 |
| Zn (%) | 1,7 | 1,8 | 1,7 | 1,5 | 2,9 | 1,4 | 2,3 | 2,0 |
| Mg (%) | 1,0 | 1,1 | 1,0 | 0,7 | 1,7 | 0,8 | 1,2 | 1,1 |
| Mn (%) | 1,2 | 1,0 | 1,5 | 2,1 | 8,9 | 5,1 | 11,8 | 10,4 |
| P (%) | 0,3 | 0,3 | 0,2 | 0,1 | 0,1 | 0,3 | 0,2 | 0,1 |
| Pb (%) | 0,8 | 0,8 | 0,9 | 0,7 | 2,4 | 1,3 | 3,3 | 3,3 |
| K (%) | 0,5 | 0,4 | 0,7 | 0,5 | 0,4 | 0,3 | 0,5 | 0,3 |
| Sn (%) | 0,3 | 0,3 | 0,3 | 0,3 | 0,7 | 0,2 | 0,5 | 0,4 |
| Na (%) | 0,1 | 0,1 | 0,2 | 0,2 | 0,3 | 0,2 | 0,1 | 0,2 |
| Ba (%) | 0,2 | 0,2 | 0,1 | 0,1 | 0,6 | 0,3 | 0,5 | 0,9 |
| Ti (%) | 0,07 | 0,09 | 0,06 | 0,03 | 0,01 | 0,09 | 0,05 | 0,03 |
| V (%) | 0,02 | 0,04 | 0,00 | 0,00 | 0,00 | 0,04 | 0,00 | 0,00 |
| Sr (%) | 0,05 | 0,04 | 0,06 | 0,07 | 0,06 | 0,04 | 0,06 | 0,08 |
| Cu (%) | 0,04 | 0,04 | 0,04 | 0,05 | 0,08 | 0,06 | 0,08 | 0,21 |
| Cr (%) | 0,02 | 0,03 | 0,02 | 0,00 | 0,00 | 0,02 | 0,01 | 0,00 |
| Ni (%) | 0,03 | 0,03 | 0,03 | 0,02 | 0,03 | 0,03 | 0,02 | 0,09 |
| Cl (%) | 0,02 | 0,00 | 0,09 | 0,00 | 0,00 | 0,00 | 0,00 | 0,00 |
| S (%) | 0,04 | 0,05 | 0,02 | 0,05 | 0,23 | 0,13 | 0,05 | 0,27 |

### 7.2.2.3 Conclusions

Based on the pXRF and XRF results, three different layers can be observed within the trench pit:

- From 0 to 0.8 m: the highest metallic element concentration is Fe with values ranging from 20 to 30 %

- From 0.8 to 1.4 m: The Fe concentration is dropping, while the concentration of the other metallic elements are increasing (especially Pb – around 4%, and Cu – around 0.04%)
- From 1.4 to 1.8 m: The Mn concentration is the highest (7-20 %), while Fe is ranging from 2 to 10 %

The Fe and Mn concentrations are not varying the same way with depth, indicating that they are not present in their alloy forms, which was expected taking into account the history of the former plant.

Regarding the chemical analysis on each separate particle size and magnetic fraction, several observations can be highlighted:

- except for iron, which concentrated in particle size fractions smaller than 600 microns, the sieving of the material do not seem to have any impact on its chemical composition.
- the magnetic fraction is mainly composed of iron (28-30%) with various concentrations of other non-ferrous metals. Manganese was more concentrated in the low-magnetic and non-magnetic fractions even if its grade (between 1 and 12% w/w) remained low compared to that observed in an ore (~40%). This observation confirmed that manganese was not in alloy with iron.

Although the analyzed samples contain various metals, their grades are too low to justify any economical interest in recovering them within the first 2 m of soil, in the particular location of the trench pit.


## 7.3 CONCLUSIONS

Based on mineral processing and chemical composition analysis, three different layers can be observed from 0 to 2 m, within the trench pit:

- From 0 to 0.8 m: the highest metallic element concentration is Fe with values ranging from 20 to 30 %.

- From 0.8 to 1.4 m: The Fe concentration is dropping, while the concentration of the other metallic elements are increasing (especially Pb – around 4%, and Cu – around 0.04%). In the center of this layer, the soil is mainly composed of fine particles and has a much higher portion of magnetic particles within which iron is the major element, revealing the potential presence of ferrous residues.

- From 1.4 to 1.8 m: The Mn concentration is the highest (7-20 %), while Fe is ranging from 2 to 10 %. In this layer, the soil is mainly composed of coarse particles (greater than 10 mm), and within its finer portions, has a lower proportion of magnetic particles, indicating a lower proportion of ferrous residues.

The Fe and Mn concentrations are not varying the same way with depth, indicating that they are not present in their alloy forms, which was expected taking into account the history of the former plant.

Although the analyzed samples contain various metals, their grades are too low to justify any economic interest in recovering them within the first 2 m of soil, in the particular location of the trench pit. Post-sampling investigations are scheduled to:

- explore the recovering potential of metals deeper in the deposit
- analyze the recovering potential of the deposit in terms of eco-catalysis.

# 8 POST-SAMPLING INVESTIGATIONS

## 8.1 SAMPLING ON SITE

No heavy machinery is allowed to enter the site. We must therefore consider the option of a lightweight auger. IXSANE proposes to study the idea of carrying out a few sampling points with an auger that can cross up to 5 m of land depending on the soil conditions.

Following the first geophysical results, it appears that the potentially most interesting soil layer starts at 2-3 m deep and up to 8-9 m deep (see deliverable DI2.2.1). It would therefore be interesting to take samples from this layer in the second sampling campaign, both on the NNW and SSE sides of the former tailing pond. Several sampling locations have been decided based on these geophysical observations

- – <u>4 samples were taken by IXANE</u> at two different locations I1 and I2 (see Figure 1): IXSANE extracted the samples using a lightweight auger that can cross up to 5 m of land depending on the soil conditions. The samples were taken at two different depth intervals: [0-0.5] m and [3-4] m
- – <u>45 samples were taken by BRGM</u> using a light core drill for depths ranging between 0 and 9.2 m at 4 different locations (see Figure 1): 11 samples for FP1 from 0 to 9.2 m, 10 samples for FP2 from 0 to 8.2 m, 12 samples for FP3 from 0 to 9.2 m, and 12 samples for FP4 from 0 to 9.2 m



*Figure 8 : a) Picture of the core drill in action in Pompey (08/2021); b) Zoom on the drilling rod with two different layers of soil.*

## 8.2 LABORATORY CHARACTERIZATION

### 8.2.1 Agronomical analysis

Four samples were taken at 2 different depths by IXANE, following the first geophysical investigations and results that indicated a conductive and chargeable anomaly, with a maximum close to the I2 sounding location, between 3 and 4 m depth :

- From 0 to 0.5 m depth: I1-A and I2-A
- From 3 to 4 m depth: I1-B and I2-B

Several agronomical parameters were measured for the four samples: pH, CaCO3 concentration, total organic carbon (C), total nitrogen (N), CEC, total phosphorous (P) and water electrical conductivity. The results are presented in Tableau 6.

Several observations can be made:

- The concentrations of C, N and P indicate a good fertility for all the samples
- The pH of all the samples is slightly alkaline
- The water electrical conductivity is higher for the deeper samples (I1-B and I2-B) than at the surface, indicating a higher ionic strength at 3-4 m, and potentially a higher concentration in metallic particles that could be soluble.

*Tableau 6 : Results of the agronomical parameters measured for the four samples taken by IXANE (see Figure 1)*

| Sample | Texture | pH$_{water}$ | CaCO$_3$ | organic C | total N | Corg/Ntotal | CEC | Electrical conductivity | available P |
|---|---|---|---|---|---|---|---|---|---|
| | | | g kg$^{-1}$ | g kg$^{-1}$ | g kg$^{-1}$ | | cmol$^+$ kg$^{-1}$ | mS cm$^{-1}$ | g kg$^{-1}$ |
| I1-A | Sand | 7,7 | | 53 | 2,80 | 18,9 | 27,1 | 0,21 | 0,135 |
| I1-B | Loamy sand | 8,2 | En attente | 28 | 0,78 | 35,9 | 19,3 | 1,57 | 0,198 |
| I2-A | Loamy sand | 8,1 | | 35 | 2,01 | 17,4 | 34 | 0,85 | 0,094 |
| I2-B | Loamy sand | 7,7 | | 27 | 1,03 | 26,2 | 37,8 | 2,13 | 0,105 |

### 8.2.2 Chemical analysis

pXRF (for the samples extracted from FP1, FP2, FP3 and FP4 locations, see Figure 1) and XRF analysis (for the I1-A and –B, and I2-A and –B samples, see Figure 1) were led at BRGM and IXANE facilities. The results for the concentration of the main metallic elements are presented in Figure 9 and Figure 10.

In Figure 9, the results of the two sounding that are the more to the North of the site, FP2 and FP3, as well as the IXANE samples (I1-A and I1-B) are shown, and compared to the pre-sampling investigations in the trench pit (fosse pXRF, fosse CTP – see part 7.2.2). Several observations can be made:

The concentrations between the pre- and post-sampling analysis for each of the elements are very consistent, except the Fe concentration that is much stronger (one decade higher) within the pit (both in the XRF and pXRF measurements) than in the soundings at the higher altitudes (close to the ground surface). As the soundings and the trench pit are not exactly at the same location, this result might be indicative of Fe concentration variations at the surface.

The Cu concentration of one sample measured by CTP (S2 at 195.3 m) is much higher than the rest of the values. This variability can be due to the low concentration of Cu (around 100 ppm) that makes its evaluation using pXRF harder.

Three main layers can be distinguished for all the metallic elements discussed here:

- **From the surface of the ground to 1 m depth** (independently of the altitude): the Mn, Pb, Zn and Sn are lower than in the second layer, but not as low as within the bottom layer. This first layer of techno-soil is the one the most subject to anthropic shuffle (uncontrolled dumping…). As the site is covered by vegetation, it is also remodeled by roots and other life forms that can potentially mobilize some of the metallic elements. These solicitations could explain the observed variations.

- **From 1 m depth to 191 m of altitude** (around 6 m depth approximately, depending on the location): the concentration of each of the metallic elements is the highest: Mn reaches $10^5$ ppm, Pb $2.5.10^4$ ppm, Zn $4.10^4$ ppm, and Sn $3.10^3$ ppm. This layer is interpreted as the deposits from the former tailing pound.

  Within this deposit layer, especially for the sounding FP3 that is at the northern end of the site, a drop in the concentrations of the different metallic elements can be observed at <u>around 193 m</u>. This can be indicative of a change in the nature of deposit, linked with the history of the plant (change in composition of the metals processed…)

- **Bellow 191 m of altitude:** the concentration of all the metallic elements drops drastically from several order of magnitude. This trend might be indicative of the bottom of the deposit, the lower concentration in metallic elements indicating the transition with a layer of natural alluvial deposits.

  It can be noted that the Fe concentration is not decreasing as drastically as the other metallic elements bellow 191 m. Although the water level is not reached yet, this element might have been mobilized by water infiltration and deposited bellow the former tailing pond deposits.
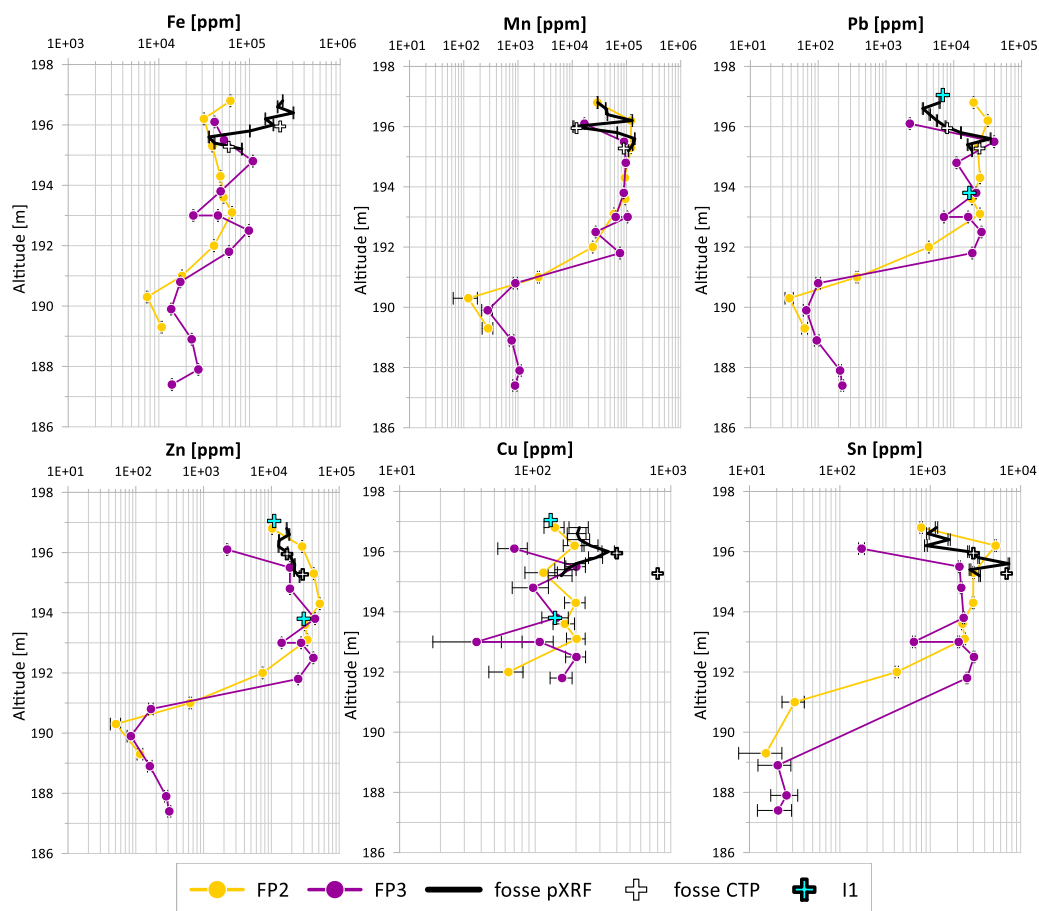


*Figure 9 : Variation of concentration of different selected chemical elements versus altitude for three soundings (FP2, FP3 and I1) at the north of the site (see Figure 1). These results are compared to pre-sampling analysis within the trench pit: (1) pXRF analysis on two profiles down to 1.8 m depth; and (2) XRF results on two samples S1 and S2 analyzed at CTP.*

In Figure 10, the results of the two sounding that are the more to the South of the site, FP1 and FP4, as well as the IXANE samples (I2-A and I2-B) are shown. The results are very similar to the ones presented for the northern part of the site. Several observations can be made in addition to the previous results:

– A good correspondence is found between IXANE analysis (for sounding I2) and pXRF analysis done at BRGM (for the two soundings FP1 and FP4)

– For FP1, the same variations versus depth can be observed than for FP2 and FP3: 3 main layers are observed at the same altitudes than FP2 and FP3, with a drop of concentration at 191 m of altitude, but the amplitude of the drop is an orderof magnitude lower than the one observed for FP2 and FP3.

– For FP4, the drop in the concentrations of metallic elements is observed at lower altitudes (at around 189 m). This observation can be linked with an increase of the thickness of the tailing pond deposits, but is quite surprising as FP4 is located closer to the edge of the former tailing pond. The thickness of the deposits is thus expected to decrease, and not increase. A finer comparison with the geophysical results will help explain this observation.
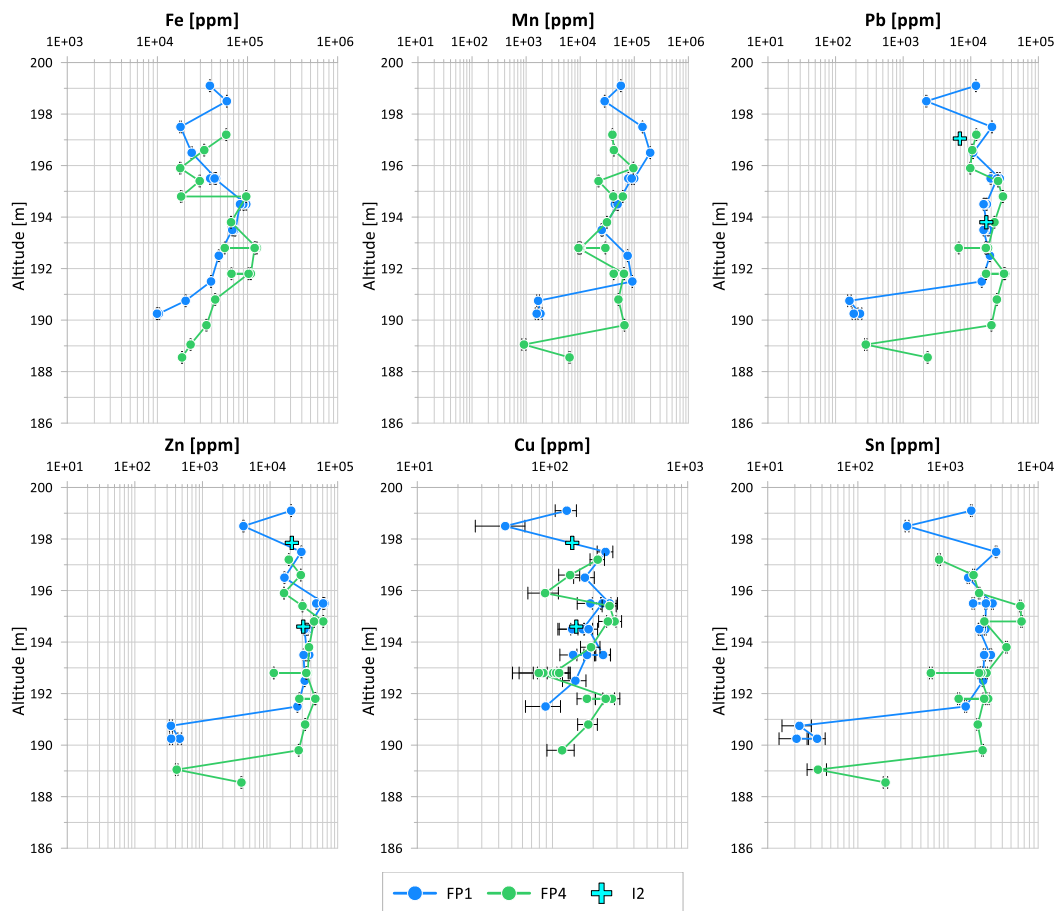


*Figure 10 : Variation of concentration of different selected chemical elements versus altitude for three soundings (FP1, FP4 and I2) at the south of the site (see Figure 1).*

## 8.3 Conclusions

The chemical characterization led both by IXANE and the BRGM on the selected samples are very consistent with the results of the pre-sampling investigations. The analysis can thus be considered as reliable and used for further interpretations.

Three main layers can be distinguished for all the metallic elements discussed here:

- **From the surface of the ground to 1 m depth** (independently of the altitude): the Mn, Pb, Zn and Sn are lower than in the layer bellow, but not as low as within the bottom layer. This first layer of techno-soil is the one the most subject to anthropic shuffle (uncontrolled dumping…). As the site is covered by vegetation, it is also remodeled by roots and other life forms that can potentially mobilize some of the metallic elements.

- **From 1 m depth to 191 m of altitude** (around 6 m depth approximately, depending on the location): the concentration of each of the metallic elements is the highest: Mn reaches $10^5$ ppm, Pb $2.5.10^4$ ppm, Zn $4.10^4$ ppm, and Sn $3.10^3$ ppm. This layer is interpreted as the deposits from the former tailing pound.

    Within this deposit layer, especially for the sounding FP3 that is at the northern end of the site, a drop in the concentrations of the different metallic elements can be observed at <u>around 193 m</u>. This can be indicative of a change in the nature of deposit, linked with the history of the plant (change in composition of the metals processed…)

- **Bellow 191 m of altitude:** the concentration of all the metallic elements drops drastically from several order of magnitude. This trend might be indicative of the bottom of the deposit. The lower concentration in metallic elements indicating the transition with a layer of natural alluvial deposits. The observed drop occurs at depths between 6 and 8 m depending on the location of the sounding. According to this set of data, the thickness of the deposit is thus lower than what was expected before the analysis (estimated at 10 m depth).

The additional agronomical analysis indicate that both the top layer (0-0.5 m depth) and the middle of the deposit layer (3-4 m depth) have good fertility rates. The water conductivity being higher in deeper layer, the fertility is a little better for the top soil. On the contrary, the concentrations of metallic elements in Cu, Hg, Pb and Zn is higher for the deeper samples. Both analyzed depth are thus interesting for eco-catalyst productions.

# 9 Recommendations

Pre- and post-sampling investigations were carried out on samples taken at different depths from a former siderurgical settling basin, potentially concentrated in metals (Fe, Zn, Mn, Pb, etc.).

The chemical analysis carried by various laboratories (CTP, IXANE and BRGM) indicate consistent metallic element concentrations, confirming the reliability of the data obtained.

Several layers can be observed looking at the concentration of the different metallic elements, with a drop at 191 m of altitude for 3 out of the 4 sampling locations FP1 to FP4, that could be indicative of the transition between the anthropic deposits and the natural alluvial deposits. It is observed at depths ranging between 6 and 8 m, which are lower than the thickness of the deposits, previously estimated to around 10 m. The total recoverable material is thus lower than expected.

Furthermore, although these samples contained various metals, their grades are still too low to justify any economic interest in recovering them.

On the contrary, the fertility rates measured are good. This type of deposits seemed thus to be more suitable for recovery by eco-catalysis.

# 10 REFERENCES

## Papers, books and reports

ANTEA, 2001a. Site du Ban-la-Dame à Frouard : Etude technique pour la mise en place d'une couverture étanche et pour l'aménagement paysager, octobre 2001 – A24803/A

ANTEA, 2001b. Site du Ban-la-Dame à Custines-Frouard : Diagnostic du bassin de décantation Sud, novembre 2001 – A25070/A

ANTEA, 2002a. Projet d'extension de Délipapier sur le site de Ban-la-Dame à Frouard : Reconnaissance de l'extension de la limite Sud de l'ancien bassin de décantation, février 2002 – A26295/A

ANTEA, 2002b. Investigations complémentaires sur le site du Ban-la-Dame à Custines-Frouard, octobre 2002 – A27912/A

ANTEA, 2003. Site du Ban-la-Dame à Custines-Frouard : Evaluation Simplifiée des Risques, mars 2003 – A29674/A

ANTEA, 2004. Ancien bassin de décantation du Ban-la-Dame à Custines/Frouard : Evaluation Détaillée des Risques pour la Santé : Scénario type promenade, mai 2004 – A32151/B

ArcelorMittal archives - file 167/092 Moselle pollution

Berrar, J.C. 2011. Le fer en Lorraine. Ed. Alan Sutton, collection Mémoire en images, Saint-Cyr-sur-Loire, France, 127 p.

BRGM, Geological map of Nancy 1/50 000, BRGM Publications.

Deliverable DT2.1.1. Mignon, B. 2021. Report on Mineral Processing lab-scale tests on samples from Pompey site (2020). 10 p.

Deliverable DI2.1.2. Kessouri, P. and Gourry, J.C. 2020. Site specific investigation plan and schedule on Pompey site (FR), 12 p.

Deliverable DI2.2.1. Kessouri, P. et al. 2021. Site specific report on geophysical survey on Pompey site (FR), 25 p.

FONDASOL, Juin 1993. Pôle d'Activités de Pompey – Frouard – Custines - Ban la Dame à FROUARD – Elargissement du chemin d'accès à la sous station EDF 63 :20 kv - Rapport d'Etude - MN ; 93/089.

Geindre, L. 1966. Pompey sous l'Avant-Garde. 80, impr. Fetzer Raon-l'Etape, France, 112 p.

Gerber, G. 2005. Pompey. Ed. Alan Sutton. Mémoires en images. 127 p.

GISFI, 2014. Note concernant les activités de recherche menées sur le bassin de décantation sud (Frouard) – GISFI – Février 2014

Huot, H., 2013. Formation, fonctionnement et évolution d'un Technosol sur des boues sidérurgiques.

IRSID. 1984. « Analyse du contexte géologique et hydrogéologique des zones des dépôts de déchets sidérurgiques de l'Usine de Pompey - étude de l'influence des dépôts sur les eaux superficielles et souterraines ». MPFE-RC 1043.

Parfitt, R. L., et A. D. Wilson. 1985. « Estimation of allophane and halloysite in three sequences of volcanic soils, New Zealand ». Catena Suppl 7: 1‑8.

Rideau, E. 1956. Essor et problèmes d'une région française. Houillières et sidérurgie de Moselle. Les éditions ouvrières, Paris. 244 p.

Rogé, M. 1982. L'Eau et la sidérurgie en Lorraine ». Thèse 3ème cycle Géographie et Aménagement, Nancy.

Schwartz, C., L. Florentin, D. Charpentier, S. Muzika, et J.L. Morel. 2001. « Le pédologue en milieux industriels et urbains. I. Sols d'une friche industrielle. » Etude et Gestion des Sols 8 (2): 135‑148.

SEMACO, 2011. Parc d'Activités de Nancy Pompey – Dossier de servitudes pour le bassin de décantation sud - Site Ban la Dame ( Frouard 54) – SEMACO 21 Nov 2011.

SNAP, 1943 - Espace Archives ArcelorMittal (plan 514-724)).

Truffaut, E. 1989. « Ferromanganèse élaboré au haut fourneau ». Techniques de l'ingénieur Elaboration et recyclage métaux (ref. article : M2445).

Truffaut, E. 2004. « La fabrication du ferro-manganèse au haut-fourneau en France, 1875-2003. Naissance, vie et mort d'un procédé industriel. » http://soleildacier.ouvaton.org/savoir/fr/DNA02-S2-W.pdf.

**Internet websites :**

Atelier Mémoire Ouvrière : website of a group of enthusiasts who bring te story of the Carreau minier from Val de Fer at Neuves-Maisons to life, http://amo.fjep.pagesperso-orange.fr/

BASOL: database on polluted or potentially polluted sites and soils calling for action by public authorities, for preventive or curative purposes, http://basol.developpement-durable.gouv.fr/

Community of communes of the Pompey basin website: http://www.bassinpompey.fr/

Géoportail: website of the National Institute of Geographic and Forest Information (IGN), http://www.geoportail.gouv.fr

Infoterre : website gathering all available BRGM data: geological maps from 1: 1,000,000 to 1: 50,000, files from the Basement Data Bank and geological logs, maps of natural and industrial risks, data on groundwater, etc., https://infoterre.brgm.fr/

# 11 APPENDIX 1: PREVIOUS CHEMICAL ANALYSIS

Data extracted from Huot (2013)

*Figure 11: Distribution of the total content of major elements (elements with an average content greater than 1 g.kg$^{-1}$ of dry soil) vs depth along the profile for 12 layers of "Technosoil" (from Huot, 2013)*
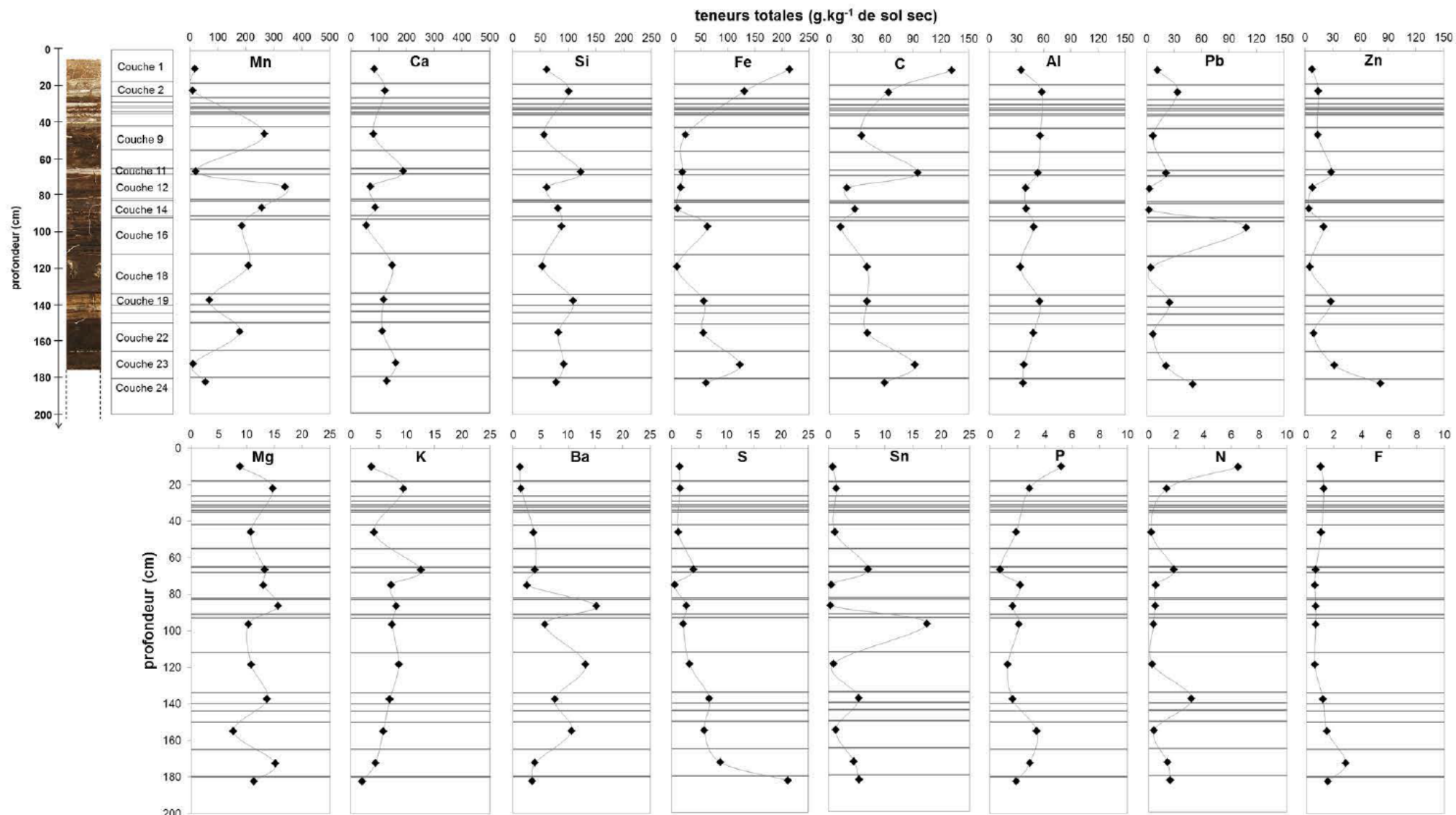
*Figure 12: Distribution of the total content of trace elements (elements with an average content lower than 1 g.kg$^{-1}$ of dry soil) vs depth along the profile for 12 layers of "Technosoil" (from Huot, 2013)*

**Tableau 7: Mineralogical phases detected using a combination of laboratory experimentations (DRX, IR spectroscopy, Mossbauer spectroscopy, SEM-EDX and TEM-EDX, Tam and Mehra-Jackson extraction methods)**

| couche | profondeur (cm) | produits allophaniques | verre silicaté | phyllosilicates/zéolites | silicates de Mn | silice – feldspaths | (hydr)oxydes de Mn non cristallisés | oxydes de Mn cristallisés | ferrihydrite | hématite | goethite | magnétite – spinelles | wüstite | calcite | carbonates ou hydroxydes de Pb | gypse | anhydrite | ettringite | barytine | Fe métallique | (hydr)oxyde de Ca | composés du Sn | cyanures |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| 1 | 0 - 18 | O | O | ox | o | x | o | | X | X | X | x | o | oX | o | | | | | x | o | | |
| 2 | 18 - 26 | O | O | ox | | | o | | X | X | X | | | X | o | | | | | x | o | | |
| 9 | 42 - 55 | | | | | | | x | | | | | | X | | | | | | | | | |
| 11 | 65 - 68 | O | O | | o | | o | x | | | | | | X | | | | | | | o | o | x |
| 12 | 68 - 82 | o | o | | | | O | ox | | | | | | oX | | | | | | | | o | |
| 14 | 82 - 91 | | | | | | | | | | | | | X | | | | | | | | | |
| 16 | 93 - 112 | o | o | | | | O | | | | | | | oX | o | | | o | | | | o | |
| 18 | 112 - 134 | | | | | | | x | | | | | | X | | | | x | | | | | |
| 19 | 135 - 150 | | | | | | | | | | | | | X | | | | | | | | | x |
| 22 | 150 - 165 | | | x | | | | | | | | | | X | | | | | | | | | |
| 23 | 165 - 180 | O | O | x | | ox | | | X | X | X | | | oX | | ox | | | o | x | | o | x |
| 24 | > 180 | | | x | | x | | | | | | | | X | | | x | x | | | | | x |

O: phase fréquemment observée en MEB-EDXS et/ou MET-EDXS ; o : phase rarement observée en MEB-EDXS et/ou MET-EDXS ; X : phase majeure détectée en DRX, spectroscopie IR et/ou en spectroscopie Mössbauer ; x : phase mineure détectée en DRX, spectroscopie IR et/ou en spectroscopie Mössbauer ; ? : phase probable

Le tableau ne présente que la liste non exhaustive des phases minérales ayant été détectées par les différentes techniques analytiques employées. Ainsi, l'absence d'une phase dans le tableau ne signifie pas forcément son absence dans la couche correspondante, d'autant plus que certaines couches (1, 2, 11, 12, 16 et 23) ont été analysées de manière plus fine que les autres.
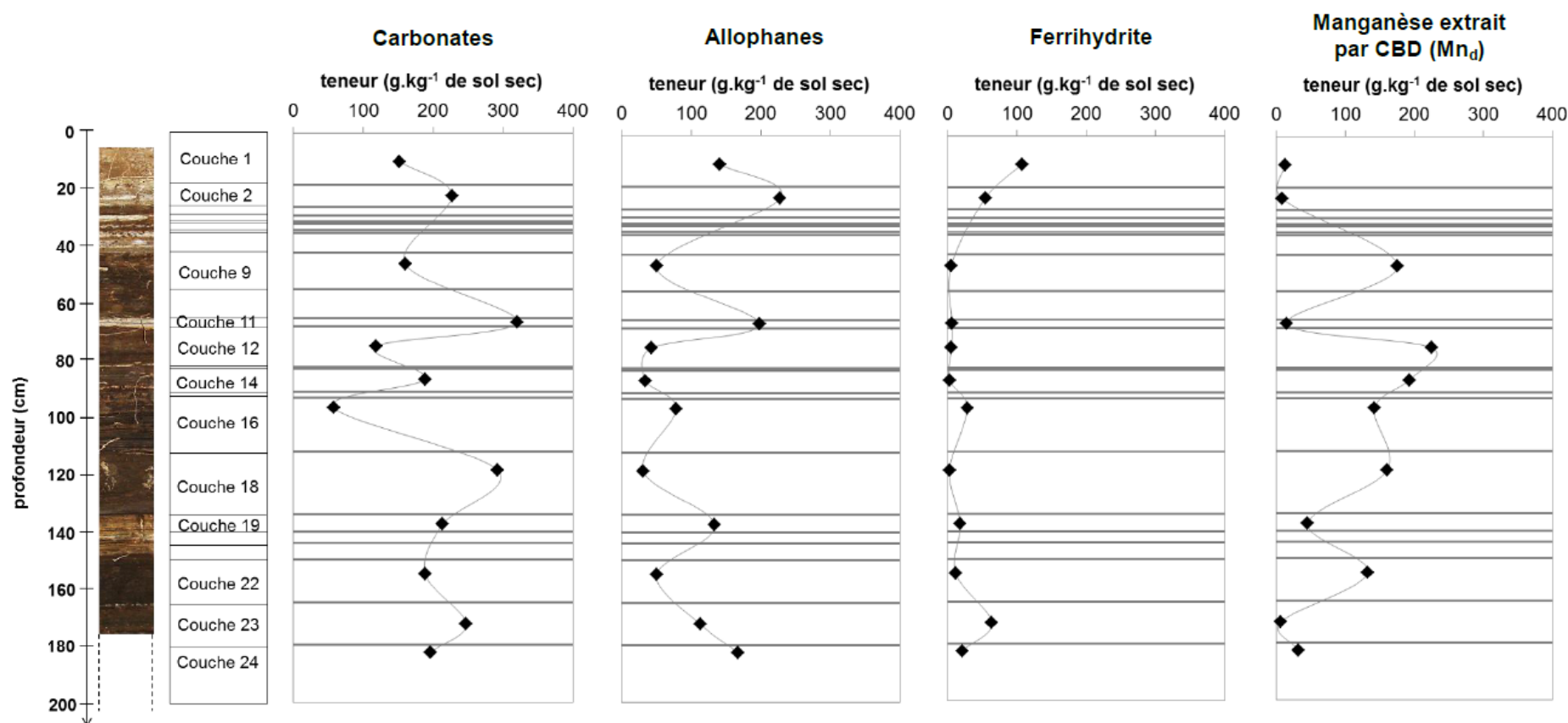
*Figure 13: Distribution of carbonate, allophanes, ferrihydrite and Mn contents extracted with CBD (Mn$_d$) according to the Mehra-Jackson method along the "Technosoil" profile (from Huot, 2013).*

*The contents are expressed in g.kg$^{-1}$ of soil dried at 105°C. The carbonate contents were determined by calcimetry. The ferrihydrite contents were estimated from the Fe contents extracted with oxalate (Fe$_o$) according to Tamm's method by the following formula: ferrihydrite = 1.7xFe$_o$ (Childs, 1985). The allophan contents were estimated from the Si contents extractedwitht oxalate according to the Tamm method (Si$_o$) by the following formula: allophanes = 7.1xSi$_o$ (Parfitt and Wilson, 1985). CBD extraction allowing the dissolution of oxides, the Mn content, extracted with CBD, can be considered proportional to the content of Mn oxides.*